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Diffractometer for high energy X-rays at the APS

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Abstract

The Basic Energy Sciences Synchrotron Radiation Center (BESSRC) has designed and built a diffractometer specialized for high energy synchrotron radiation ($E > 60$ keV) at the Advanced Photon Source (APS). The diffractometer, which is installed at the elliptical multipole wiggler, uses linearly polarized light (U. Rütt et al., Proc. SPIE 3348 (1998) 132.). The instrument is a triple-axis diffractometer allowing high resolution measurement in two dimensions of the reciprocal space. As opposed to the other diffractometers for high photon energies at HASYLAB (Germany) and ESRF (France) (R. Bouchard et al., 5 (1998) 90; K.-D. Liss et al., J. Synchrotron Rad. 5 (1998) 82), this diffractometer utilizes the vertical scattering plane to take full advantage of the small vertical divergence of the beam and to allow horizontal focusing of the broad beam from the wiggler without disturbing the resolution of the instrument. The instrument is designed to carry heavy sample equipment up to a weight of 200 kg, while maintaining high resolution and low background. First tests have been done to check the overall performance, stability, background and resolution of the diffractometer.

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Experiments using high energy synchrotron radiation ($E > 60$ keV) gain from a large mean free beam path of the photons in materials. At high photon energies momentum transfers of $Q > 45\,\text{Å}^{-1}$ are easily achievable. In addition, the correction factors due to polarization, absorption and extinction are small in general [1–3]. For magnetic scattering, the cross-section is proportional to the square of the spin component [4], which allows the determination of the pure spin contribution to the magnetic moment [5]. The high penetration into material and the high momentum transfers make high energy photons comparable to neutron experiments, but with the high resolution and high flux of modern X-ray instruments at synchrotron radiation sources.

A triple-axis diffractometer consisting of monochromator, sample and analyzer provides high q-space resolution in two dimensions. The monochromator crystal determines the scattering plane and the energy bandwidth. The analyzer rotation determines the observed lattice spacing at the

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sample and allows studies in two dimensions of the reciprocal space. For three perfect Si 2 2 0 crystals at 100 keV photon energy the q-space resolution defined by the FWHM is 10^{-5} Å^{-1} and 2×10^{-4} Å^{-1} perpendicular and parallel to the reciprocal lattice vector. The resolution perpendicular to the scattering plane is determined by the angular acceptance of the detector. Because of low absorption and small Bragg angles all crystals are used in transmission geometry. This allows studies of large samples (up to several mm).

The diffractometer can also be used for powder diffraction gaining from good averaging over a large sample volume as well as for studies of pair distribution functions in amorphous or powdered materials, which take full advantage of large momentum transfers [2].

The Basic Energy Sciences Synchrotron Radiation Center (BESSRC) has built a triple axis diffractometer for high energy photons at the elliptical multipole wiggler (EMW) with a critical photon energy of 32 keV at the Advanced Photon Source (APS) [6]. A pre-monochromator diffracts horizontally at a Bragg angle of $\theta=1.9^\circ$ into the station. The pre-monochromator is at a distance of 32 m from the source and 22 m away from the diffractometer. If an annealed silicon 2 2 0 crystal (10 mm wide) with a mosaicity of 10 arcsec [7] is used in transmission geometry, the beam spot of 98 keV photons at the diffractometer is up to 3.5 mm high and 2.5 mm wide with an intensity of 2×10^{12} photons/sec and an energy resolution of $\Delta E/E=0.01$. The beam size and energy band width can be adapted by a slit directly behind the pre-monochromator. For high resolution experiments only 0.06 mrad of the beam would be scattered providing an energy bandwidth of $\Delta E/E=0.002$. The beam spot in the hutch is hereby reduced to 1 mm horizontally with an intensity of 4×10^{11} photons/s.

The photon energy can be changed by the choice of the lattice spacing of the pre-monochromator. At the moment annealed Si 1 1 1, Si 2 2 0 and Si 3 1 1 crystals are available scattering 60, 98 and 115 keV into the hutch, respectively.

As opposed to the three other dedicated triple crystal diffractometers for high energy photons [2,3], this diffractometer is operating in the vertical scattering plane, see Fig. 1. The difficulties of this geometry were to allow for heavy sample environment while maintaining the angular stability better than 0.5 arcsec required for high resolution experiments. The advantages are the flexibility to use it for high momentum transfers as well as for high resolution experiments and the option of focussing in the horizontal scattering plane while taking advantage of the narrow vertical beam divergence for high energy resolution.

The diffractometer has four towers for monochromator, sample, analyzer and detector. The first three are placed on a common optical table (Newport RS4000, 3.6 m long, 1.5 m wide and 203 mm thick). The monochromator and analyzer towers are 610 mm wide, the sample tower 650 mm wide. All three towers can be moved along the table on a rail system. Monochromator and analyzer tower are equipped with a Huber 420 goniometer for the θ-rotation and a Huber 408 and Huber 5202.5 circle segment for the crystal alignment. On the analyzer tower the θ-rotation is controlled by a Heidenhain encoder (0.2 arcsec accuracy and 0.04 arcsec step resolution). The goniometer can be translated by a vertical (z) rail system (900 mm travel). An additional horizontal (x) rail system below this tower gives the option to scatter horizontally. The sample tower has a heavy duty z-translation as the base of the rotation tables. The θ-rotation is realized by a Huber 430 with counter point which supports the following tables: a Huber 420 as "psi"-rotation, a circle segment made by Huber with an angular range of $\pm10^\circ$ providing the χ-rotation, a Schneeberger z-translation (±12 mm) and x-translation (±50 mm). The translations allow the choice of different sample volumes in large crystals. Optional, an additional Huber 420 as φ-rotation parallel to the θ-rotation or a κ-arm at an angle of 60° can be mounted. The sample tower can carry heavy loads up to 200 kg. All three θ-rotations of the diffractometer are monitored by tilt sensors to control unwanted angular motions (better than 0.1 arcsec).

The detector tower is located behind the optical table. The distance between detector tower and optical table can be varied between 0 and 1.5 m. A x-translation, which allows the use of the

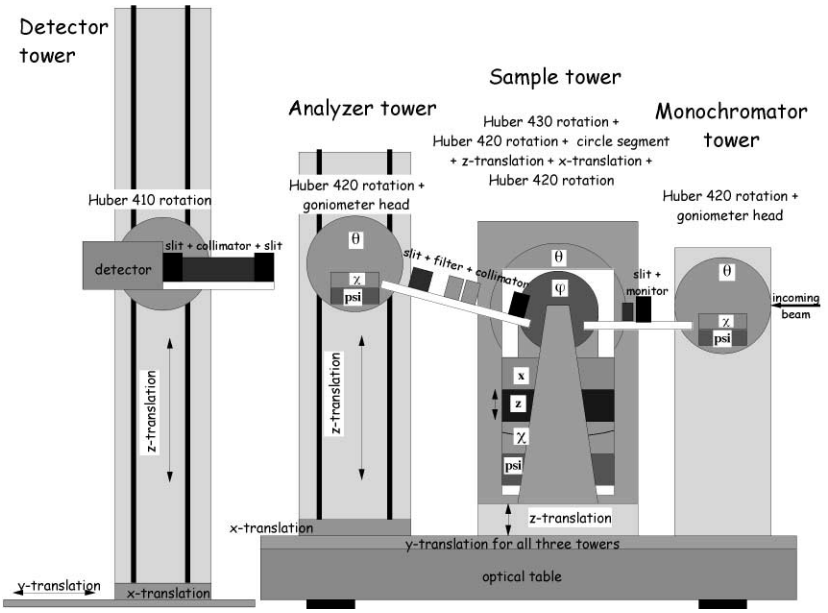

Fig. 1. Sketch of the diffractometer. The three towers for the crystals are mounted together on an optical table (3.6 m × 1.5 m). The detector is placed behind the diffractometer on a tower that can be translated in all three directions. The detector is kept parallel to the incoming beam by a rotation. Optical rails between the towers allow mounting of slit systems, beam collimators and beam monitors. For a more detailed description see text.

horizontal scattering plane, is 2 m long, the z-translation has a travel range of 1.5 m. The detector is kept parallel to the incident beam with a Huber 410 rotation table supported by a counterpoint. The detector is a single element germanium solid state detector with a crystal 18 mm thick and 16 mm in diameter. Its energy resolution is better than 700 eV at 122 keV with 1 μs shaping time.

The utilization of monochromator and analyzer crystals are optional. They are only needed for high resolution experiments. An analyzer with a broad mosaicity, e.g. annealed silicon, can also function like a narrow slit to suppress background, even if good two dimensional resolution is not required.

For experiments with powders or amorphous materials studying pair correlation functions the sample can be placed on the analyzer tower. In this case a x- and z-translation are available for an easy sample alignment. Sample and detector can be as close as 650 mm. A momentum transfer of 48 Å^{-1} can be reached using 115 keV photon energy.

Between the four towers are optical rails guided automatically with the z-translations of the towers. On these rails slit systems, collimators and beam monitors can be placed. Four motorized slits are available. Two of the slits are especially equipped with 5 mm tungsten and 10 mm tantalum blades, respectively. The brass collimators have holes for the beam path of 10 mm or 20 mm in diameter. As beam monitors silicon photon diodes (10 mm × 10 mm × 1 mm thick) are used in transmission geometry.

As sample surroundings a helium flow cryostat and a displex are available for low temperature work at the moment. In general no special windows are necessary because of the small absorption of the high energy photons, so that various laboratory equipment could be used.

The user interface to the diffractometer is implemented in the program package Igor running on Macintosh. An UB-matrix can be used to perform scans in the reciprocal space. Motors and scalers are driven by a VME-crate with EPICS system.

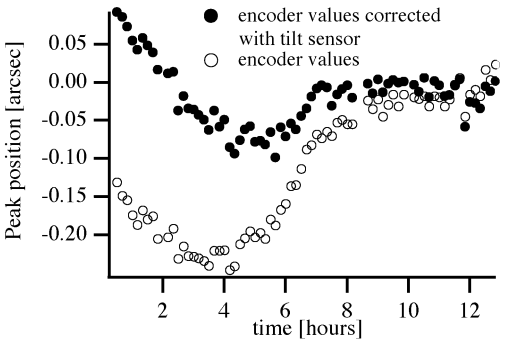

Fig. 2. Test of the angular stability. Perfect Si 2 2 0 crystals were mounted on the monochromator and analyzer tower. The analyzer crystal was scanned every 10 min with its rotation controlled by a Heidenhain encoder. In addition the stability of the towers was monitored by tilt sensors. The peak position of the analyzer reflection was stable <0.3 arcsec over 12 h. After the correction for the motion of the towers from the tilt sensor, the stability was better than 0.15 arcsec.

To test the stability of the instrument, perfect Si 2 2 0 crystals were mounted on the monochromator and analyzer tower. The analyzer crystal was scanned over 12 h, with a measurement done every 10 min to check for changes in the angle. As shown in Fig. 2, the stability was better than 0.15 arcsec, when the tilt sensor were taken into account. Over long term the stability increased, supposedly because the instrument was undisturbed during the measurement.

A further test of the reliability of the diffractometer was the determination of absolute lattice constants with a reference crystal [8]. For this experiment, three perfect Si 2 2 0 crystals were used as monochromator, reference sample and analyzer crystals. In addition, the sample, here Ge(2 2 0), with lattice spacing d_{sample} was placed next to the reference crystal with a well known lattice spacing $d_{\text{ref}} = 1.92011$ Å on the sample stage. Using transmission geometry, the incident photon wavelength $\lambda = 0.12588$ Å was calculated from the rotation angle between the Si(2 2 0) and Si($-2\ -2\ 0$) reflection of the analyzer crystal. From the difference in the angle $\Delta\omega = 542.6$ arcsec of the analyzer crystal for the reflections from sample and reference crystal, the lattice constant of the sample can be calculated by $d_{\text{sample}} = d_{\text{ref}}/(1-\Delta\omega \cdot d_{\text{ref}}/\lambda) = 2.00038$ Å ($d_{\text{Ge(2 2 0)}} = 2.00023$ Å in literature). Assuming we can measure $\Delta\omega$ better than 1 arcsec, an absolute lattice spacing can be determined to a precision 0.01% of the lattice spacing of the reference crystal with λ in the order of 0.1 Å.

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